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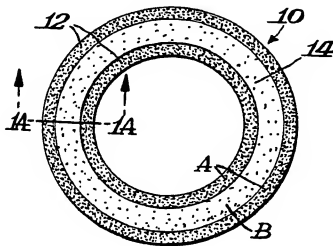
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<p>(21) International Application Number: PCT/US89/02302 (22) International Filing Date: 25 May 1989 (25.05.89) (30) Priority data: 198,532 25 May 1988 (25.05.88) US (71) Applicant: W.L. GORE &amp; ASSOCIATES, INC. [US/US]; 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714 (US). (72) Inventor: SNYDER, Ritchie, Alan ; 108 Montchanin Road, Wilmington, DE 19807 (US). (74) Agents: MEYER, Dena et al.; W.L. Gore &amp; Associates, Inc., 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714 (US).</p>		<p>(81) Designated States: AT (European patent), AU, BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>

(54) Title: GASKETING PRODUCTS AND MANUFACTURE THEREOF



(57) Abstract

A rigid and compressible gasket is provided consisting of a porous polymeric material that is of variable density across its cross-section and is capable of providing a seal over a range of temperatures from -450° F to over +600° F. A preferred porous, polymeric material is expanded porous PTFE.

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## GASKETING PRODUCTS AND MANUFACTURE THEREOF

BACKGROUND OF THE INVENTION1. Field of the Invention

This invention relates to a gasketing material having variable density across its cross-section, functional over a wide temperature range and inert to most chemicals and capable of maintaining structural integrity, rigidity, and sealability. The invention, specifically provides an insertable gasket comprised of expanded polytetrafluoroethylene (hereinafter PTFE) and the manufacture thereof.

2. Description of the Prior Art

There exist many applications where a gasketing material is required to be rigid such that it can be placed in a specified location with a specific shape and not deform under its own weight. An example of a rigid gasket is one placed between two adjacent pipe flanges.

A gasket having structural rigidity is also desirable for robotic applications or with the use of automatic equipment. The gasket must be structurally rigid so that when handling, it does not deform thereby rendering itself defective. One drawback of such gaskets however is that they are not easily compressible and thus fail to adequately seal any rough surface.

Gaskets are also available that are highly compressible so that they can conform and seal irregular shapes and surfaces. These types of gaskets suffer however in that they are not rigid and thus deform under their own weight.

As such, commercially available gaskets are categorized into two broad groups, namely:

1. Very rigid and stiff requiring high compressive loads during service in the range of as high as 10,000 psi to acquire a desired seal. Gaskets falling into this category include for example: Compressed asbestos, metal-jacketed gaskets, and spiral wound gaskets and;

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2. Highly compressible gaskets that tend to be non-rigid, but easily conformable to irregularities on rough or smooth surfaces using low compressive loads. Gaskets falling into this category include for example: Joint Sealant such as that commercially available from W. L. Gore & Associates, Newark, DE and Liquid Room Temperature Vulcanization (RTV) silicone gaskets.

In certain applications, such as with glass-lined pipes, glass-lined vessels, and other glass-lined entities with irregular bolt patterns, high compressive loads cannot be achieved without distortion or deformation of the workpiece. It is very difficult with insertable gaskets presently available to achieve the desired seals without damaging the workpiece. Typically, one type of gasket is not functional over a range of temperatures from -450°F to +600°F and simultaneously over a wide range of compressible loads. A multiplicity of gaskets comprised of varying materials would have to be used to provide sealing capabilities over the wide temperature range and wide compressive loads.

#### SUMMARY OF THE INVENTION

A rigid and compressible gasket is described consisting of a porous polymeric material that has density in one region greater than the density in another region of the gasket. The gasket also provides capability of providing a compressible seal over a range of temperatures from -450°F to over +600°F. The polymeric material may also contain a filler such as carbon, graphite, silica, asbestos, pigments or other materials. A preferable polymeric material is porous PTFE. A preferable embodiment of this invention is an insertable gasket and may be of any shape. The gasket may also be provided with holding means such as tabs or bolt holders for easy insertion into a workpiece.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a topview of the invention in the form of an insertable gasket.

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Figure 1a is an enlarged cross-sectional view of the invention taken along line 1A-1A of Figure 1.

Figure 2 is a topview of a second embodiment of the invention.

5 Figure 2a is an enlarged cross-sectional view of the second embodiment taken along line 2A-2A of Figure 2.

Figure 3 is a pictorial view of a third embodiment of the invention.

Figure 3a is a top plan view partially broken away to show the densified and undensified regions of the third embodiment.

10 Figure 4 is a top plan view of the outershell die used to make the invention.

Figure 4a is a cross-sectional view to show the die and material placement within the die taken along line 4A of Figure 4.

15 Figure 5 is a bottomview of the inner shell die used to make the invention.

Figure 5a is a side elevational view partially broken away to show the inner die.

20 Figure 6 is a schematic sideview of the test equipment used in Example 2.

Figure 7 through 15 are graphs of sealability test results comparing the inventive gaskets with commercially available gaskets as described in Example 2.

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#### DETAILED DESCRIPTION OF THE INVENTION

This invention relates to the manufacture and article made therefrom of a gasket comprised of polymeric material having varying density. The porous polymeric material may be comprised of a porous polyolefin such as porous polypropylene. The most preferred polymeric material is porous polytetrafluoroethylene (PTFE) made in accordance with U.S. Patent Nos. 4,187,390 and 3,597,566. U.S. Patents 4,187,390 and 3,597,566 are both herein incorporated by reference. Alternatively, porous PTFE made by any other method is also suitable. Also, the porous PTFE may be either heat treated to amorphously lock the material or may not be

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heat treated at all or partially heat treated. In particular, the inventive gasket is comprised entirely of porous PTFE wherein predetermined areas of the gasket are densified or are densified to a greater degree than other areas resulting in a product that has a combination of high density and low density regions. By densifying the gasketing material in a selected pattern, the overall gasket is provided with sufficient rigidity and self-integrity so that it does not deform under its own weight. At the same time however, the undensified regions of the gasketing material provides for the gasket to be highly compressible and thus provides for good sealing at relatively low sealing forces.

The polymeric material used in this invention may also contain fillers such as graphite, carbon black, asbestos, silica, pigment, mica, titanium dioxide, glass, potassium titanate, dielectric fluid, and polysiloxane.

The inventive gasket may be used in a variety of applications to seal two adjoining surfaces whether the article be placed as a permanent fixture to the workpieces or whether it be used as a replaceable or insertable article so that when a gasket needs to be replaced, the inventive article may be inserted or replaced.

The shape of the gasket may be circular, rectangular, or have any other geometric shape. Alternatively, the gasket may be in the shape of an O-ring (i.e. closed loop) or be a long strip.

The gasket may also be provided with holding means such as tabs or bolt holders for easy insertion in a workpiece. These holding means may be attached by mechanical means, adhesives, bonding agents or any material causing the holding means to be adhered to the gasket.

The invention may best be understood by reference to the accompanying drawings. Figure 1 is a graphical topview of one embodiment of the invention as an insertable gasket 10 wherein the gasket is in the shape of an O-ring.

The heavily dotted region 12 of the gasket 10 shows the densified region of the gasket in which the polymeric material has more than about twice the density of the center region 14 of the gasket. The pattern of the densified segment of the gasket may

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vary so that it covers substantially the overall surface area or in the alternative, covers only a minimal portion of the surface area.

5 An enlarged cross-section of the gasket is seen in Figure 1a. Gasket 10 is comprised of expanded porous PTFE wherein the edges 12 have a density greater than that of the central portion of the gasket 14. Such a gasket with a pattern of variable density regions provides for structural rigidity in the high density regions and allows low density regions to retain areas of high  
10 compressibility and high sealing characteristics thereby rendering itself an excellent material for insertable gaskets. The regions of high density provide rigidity to the gasket such that it will not deform under its own weight but rather will retain its original shape.

15 The high compressibility characteristics permit low density expanded PTFE to conform easily to irregular surfaces and to compress into a thin ribbon under pressure. This results in maximum sealing with the compressible material.

20 Figure 2 is a topview of a second embodiment of a gasket 20 showing a densified region 24 in the center of the O-shaped ring depicted by heavy dotting and having the polymeric material of low density at the inner and outer edges 22.

Alternative to the circular shaped gaskets previously described, Figure 3 shows a rectangular gasket wherein one region  
25 34 is comprised of a polymeric material having a density greater than the density in another region 32. Figure 3a shows a top plan view partially broken away to show a gasket wherein one region 34 has a density greater than that of region 32.

To construct a gasket with varying density, reference is made  
30 of Figure 4. A rod of porous polymeric material 46 is fitted into the well 42 of die 40. The volume of the rod 46 is greater than that of the die so that the edges of the rod overlap the horizontal edges 44 of the die as shown in Figure 4a.

35 A second inner shell die 50 shown in Figure 5 is placed on top of the outer shell 40 containing the polymeric rod. The inner shell 50 should fit snugly within the outer shell 40 so to be in

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contact with the polymeric rod 46. A well 52 is also molded in die 50. In this case, pressure greater than 9000 pounds (2100psi) was applied to the inner and outer shell, thereby selectively densifying regions of the polymeric material. Figure 5a shows a side elevational view partially broken away to show the inner die and well. The cross-hatched area of 54 in Figure 5a fits directly over and covers the surface of the cross-hatched area of Figure 4a.

An alternative and more preferred method is to use the dies as described above with a combination application of heat and pressure. Using this method, the die containing the polymeric material is heated by a heating means to above room temperature, preferably so that the die is heated to a temperature of 400°C for about 15 minutes. Simultaneous with the heating step, pressure is applied. For a die heated to about 400°C, minimum pressure of about 1130 psi is necessary, although higher pressure loads may be applied.

The resulting selectively densified polymeric may be used as is or may be further modified into a desired shape. A particularly useful shape for the material is a closed loop or O-ring, wherein the ends of the rod are joined together under pressure and/or heat, or with the use of an adhesive, bonding agent or other means available for joining polymeric materials.

Preferably, the seam where the two ends are joined remains the same volume as the rest of the gasket so that there is no discontinuity in shape. One method to maintain the volume is to taper the ends to be joined so they fit snugly together without overlap and thus without an increase in diameter and volume.

The resulting selectively densified gasket has patterned regions of high density that provide rigidity in that the gasket can support itself under its own weight and thus provide for good handling characteristics. Coincidentally, the regions of the gasket that had not been densified provide for greater compressibility in the workpiece so as to form a tight seal. The preferred polymeric material for these gaskets is comprised of porous PTFE and thus makes the gasket chemically inert and capable of performing over a wide temperature range, typically from -450°F to about +600°F.



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In addition to the shapes described above, the gasket may be of a variety of shapes. They may also be fitted with items to assist in the placement within the workpiece, for example tabs, bolt holes, or adhesives. The following examples are intended to illustrate but not limit the present invention.

#### EXAMPLE 1

A two inch diameter insertable pipe flange gasket comprised of expanded porous PTFE was made. An outershell die was first manufactured using ANSI B16.5-1981 code for class 150 flange and  
10 Figure 4. The dimensions of the die well R was 0.11 inches.

A rod approximately 13 inches in length of porous PTFE having a 3/8 inch diameter was made in accordance to the teachings of U.S. Patent 4,187,390 and was not heat treated to above its  
15 crystalline melt point. The rod was inserted into the well 42 of the outer shell die 40 shown in Figure 4. The ends of the rod were tapered to an angle of 30° each, so that when joined together, there existed a smooth transition in cross section. The  
20 inner shell 50 also having a die well, was then placed on top of and into the outer shell 40 containing the PTFE rod so that the inner shell covered in its entirety the porous PTFE rod and had a larger volume of PTFE in the well region than at the edges of the shell.

The die combination was then placed in a hydraulic press at an ambient temperature of 24°C and a force of 2050 psi across 4.35  
25 in.<sup>2</sup> or 8900 lbs.f was applied perpendicular to the plane of the die. The load was applied for one minute.

After one minute, the load was released from the die and the die assembly was removed from the press. The shells of the die  
30 were separated and the gasket of variable density was removed.

For each region of the resulting gasket (i.e. densified region and undensified region), density measurements were taken on four random samples.

The dimensions (length, height, and width) was computed for

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each sample by using a vernier caliber. The volume for each sample was calculated and the weight of each sample using an analytical balance was measured. Density was calculated by the formula  $P = \text{Wt.}/\text{Vol.}$  An average density was calculated for each region. All density measurements are summarized in Table 1. The specific regions are identified by letter in Figure 1.

EXAMPLE 2

An insertable gasket was made from a rod of expanded porous PTFE similar to that described above. The die well was 0.385 inches wide with a radius R of .120.

In the manufacture of this gasket, the rod material was placed in the outer shell, with the inner shell on top. The die combination was then pre-loaded with a 2.5 psi (or 11 pounds) force applied perpendicular to the die for 10 seconds to prevent shrinking when heat treated. The die assembly was then placed in an air-recirculatory oven set at a temperature of 400°C after which a 1130 psi (or 4920 lbs.) load was applied to the die assembly for one minute. Density results of Example 2 are also summarized in Table 1. Reference again should be made to Figure 1 to identify the regions.

TABLE 1Density g/cc

	<u>Region A</u>	<u>Region B</u>	<u>Region A</u>
Example 1	.6-.7	1.5-2.1	.6-.7
Example 2	.5-.6	1.2-1.3	.5-.6
Example 3	.7	1.8	.7

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## EXAMPLE 3 - SEALABILITY TEST FOR GAS TESTING

The sealability of the inventive gasket as well as the sealability of presently commercially available gaskets were tested using a modified version of ASTM F37-82. The apparatus used for testing can best be understood by reference to Figure 6. The sample gasket 70 made in accordance with Example 1, was placed on the bottom platen 71 of which the square area of the gasket was measured to be 5.5 sq. in. The bottom platen 71 was raised to meet the top platen 72. The means for raising the lower platen for this experiment was a hydraulic cylinder.

An initial load of 5000 lbs. across the 5.5 square inch area was first applied. An internal air pressure of 30 psi was then applied to the gasket by opening valve 73 while valve 74 was closed. The water level in the manometer varies with leakage through the gasket. After 30 minutes, the increase in water height was noted and recorded. Valve 74 was opened and the system was allowed to equilibrate.

This test procedure was repeated and involved increasing the applied load pressure at 1000 psi increments. Thirty minutes after each increased increment, manometer measurements were taken. Test results were terminated when the water rise was less than 1/32 inch per 30 minute time period. Comparative tests using other commercial gaskets of similar dimensions were also conducted under the same conditions. Results of all tests are presented in Figures 7-15. Reviewing the results, the compressed asbestos, EPDM, SBR, Neoprene rubber, nitrile, Gylon® and Teflon® require loads greater than that of the inventive gasket to obtain a leak rate below 1/8 inch water rise during a 30 minute time period.

The inventive gasket obtains a leak rate of less than 1/16 water rise at 11,000 psi for a 30 minute time period.

EXAMPLE 4 - FILLED INSERTABLE GASKET

An insertable O-ring gasket was made comprising 80% porous PTFE by weight and 15% by weight of graphite.

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5 A rod of porous PTFE not heat treated to above its crystalline melt point and filled with graphite was placed into the outershell die described above. The rod of material was wrapped twice within the shell so that two layers of porous PTFE-graphite filled the well. An inner shell was placed within the outer so as to contain the graphite filled porous PTFE and a 15,000 lb. load was then placed over the die for one minute.

10 The resulting structure was rigid in certain areas where they were selectively densified and compressible in others.

Results of the densified and undensified regions are found in Table 1.

15 It will be apparent to those skilled in the art that various modifications and variations can be made in the processes of the present invention. Thus, it is intended that the present invention cover the modifications and variations of this invention provided that they come within the scope of the appended claims and their equivalents.

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What is claimed is:

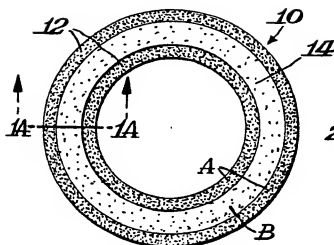
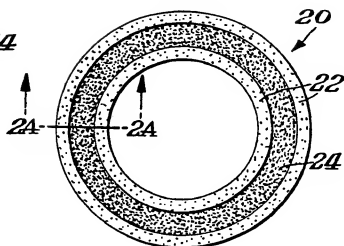
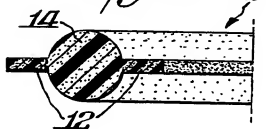
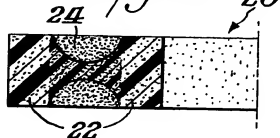
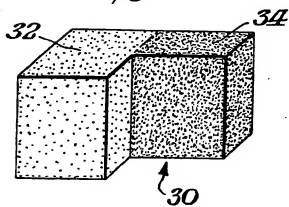
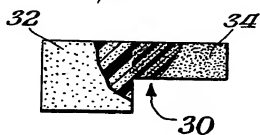
1. A rigid and compressible gasket consisting of a porous polymeric material having regions wherein the density of the material is greater in one region than the density in another region.
2. A rigid compressible gasket as described in Claim 1 wherein said porous polymeric material is porous PTFE.
3. A rigid compressible gasket as described in Claim 2 wherein said porous PTFE is heated above its crystalline point.
4. A rigid compressible gasket as described in Claim 1 that is an insertable gasket.
5. A rigid compressible gasket as described in Claim 1 in the shape of a rod.
6. A rigid compressible gasket as described in Claim 1 in the shape of a closed loop.
7. A rigid compressible gasket as described in Claim 1 wherein the porous polymeric material contains a filler.
8. A rigid compressible gasket as described in Claim 7 wherein the filler is graphite.
9. A rigid compressible gasket as described in Claim 7 wherein the filler is carbon black.
10. A rigid compressible gasket as described in Claim 7 wherein the filler is asbestos.
11. A rigid compressible gasket as described in Claim 7 wherein the filler is silica.
12. A rigid compressible gasket as described in Claim 7 wherein the filler is pigment.
13. A rigid compressible gasket as described in Claim 7 wherein the filler is mica.
14. A rigid compressible gasket as described in Claim 7 wherein the filler is titanium dioxide.
15. A rigid compressible gasket as described in Claim 7 wherein the filler is glass.
16. A rigid compressible gasket as described in Claim 7 wherein the filler is potassium titanate.

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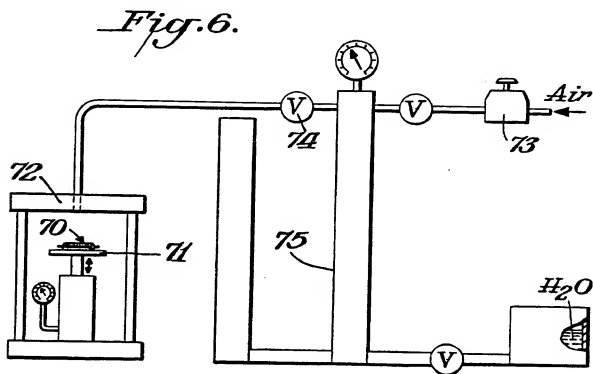
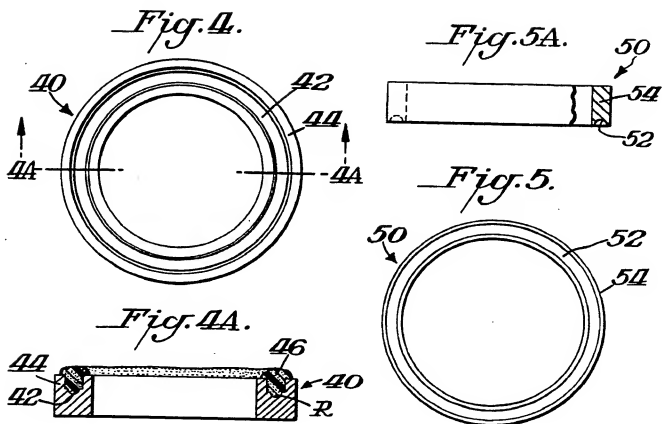
17. A rigid compressible gasket as described in Claim 7 wherein the filler is dielectric fluid.
18. A rigid compressible gasket as described in Claim 7 wherein the filler is polysiloxane.
- 5 19. A rigid compressible gasket as described in Claim 1 wherein the porous polymeric material is porous polyolefin.
20. A circular rigid compressible closed loop shaped gasket consisting essentially of expanded porous PTFE having a density in a section of said closed loop greater than a  
10 density in another section of said loop.
21. A circular rigid compressible closed loop shaped gasket as described in Claim 20 wherein inner and outer diameter edges have a density greater than the density in the middle section of said loop.
- 15 22. A circular rigid compressible closed loop shaped gasket as described in Claim 20 wherein a middle section of the loop has a density greater than the density at inner and outer diameter edges.
- 20 23. A method of making a rigid and compressible gasket comprising inserting a porous polymeric material into a die and selectively compressing regions of said polymeric material so that one region has a density higher than another region.
24. A method of making a rigid and compressible gasket as described in Claim 23 further comprising joining ends of said  
25 polymeric material to form a closed loop.
25. A method of making a rigid and compressible gasket as described in Claim 24 wherein inner and outer edges of the polymeric material are selectively compressed so that the inner and outer edges have a density higher than an interior  
30 region of said gasket.
26. A method of making a rigid and compressible gasket as described in Claim 24 wherein interior regions of the polymeric material are selectively compressed so that the interior region has a density higher than inner and outer  
35 edges.

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27. A method of making a rigid and compressible gasket as described in Claim 23 comprising using porous PTFE as the porous polymeric material.
- 5 28. A method of sealing the space between two surfaces comprising separating said surfaces, inserting a rigid and compressible gasket of a polymeric material that has been selectively compressed so that one region of said gasket has a density higher than the density in another region, and compressive loading the surfaces.
- 10 29. A method of sealing the space between two surfaces as described in claim 28 wherein the polymeric material of the gasket has a density greater at inner and outer edges than the density of the inner region.
- 15 30. A method of sealing the space between two surfaces as described in Claim 28 wherein the polymeric material of the gasket has a density greater in the inner region than the density at the inner and outer edges.
- 20 31. A method of sealing the space between two surfaces as described in Claim 28 wherein the gasket is in the shape of a closed loop.
32. A method of sealing the space between two surfaces as described in Claim 28 wherein the polymeric material is porous PTFE.

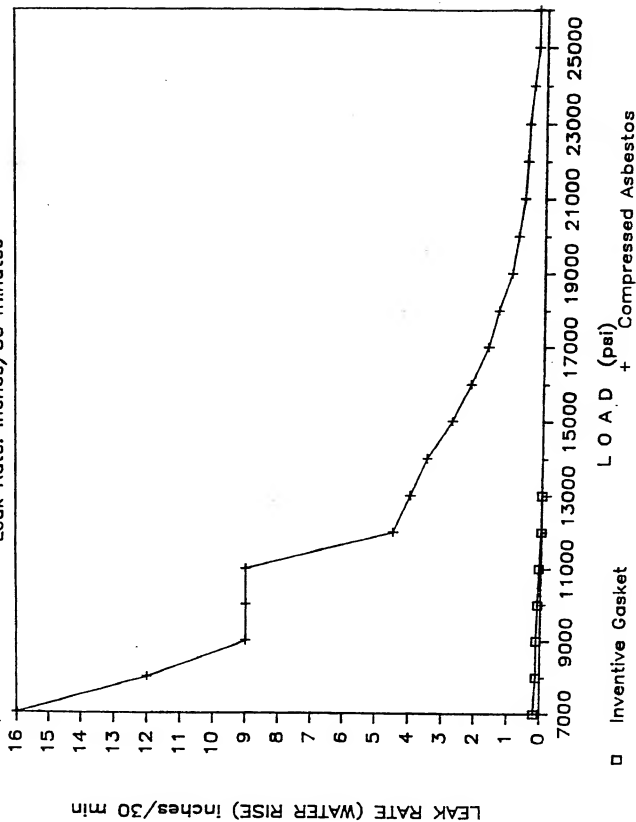
*Fig. 1.**Fig. 2.**Fig. 1A.**Fig. 2A.**Fig. 3.**Fig. 3A.*





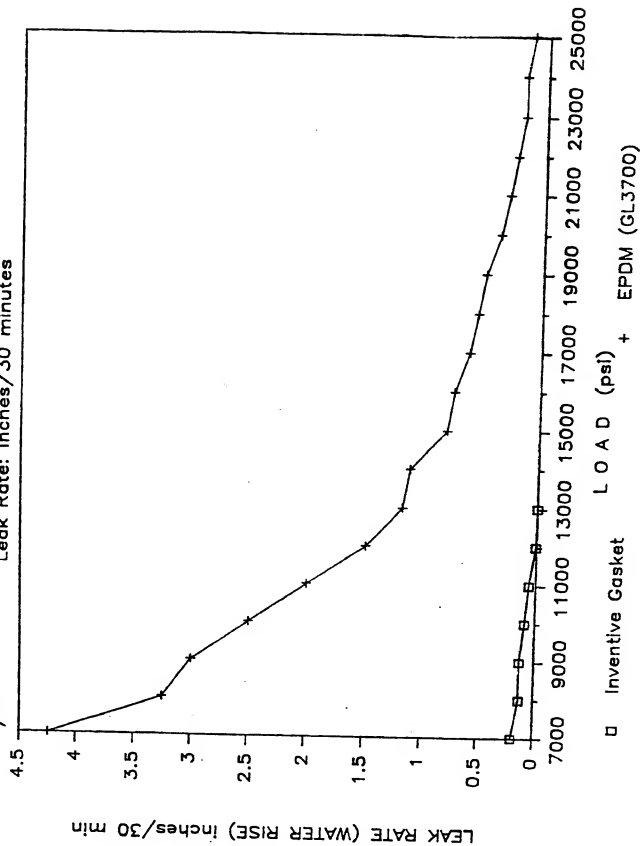
*Fig. 7.* SEALABILITY TEST

Leak Rate: inches/30 minutes



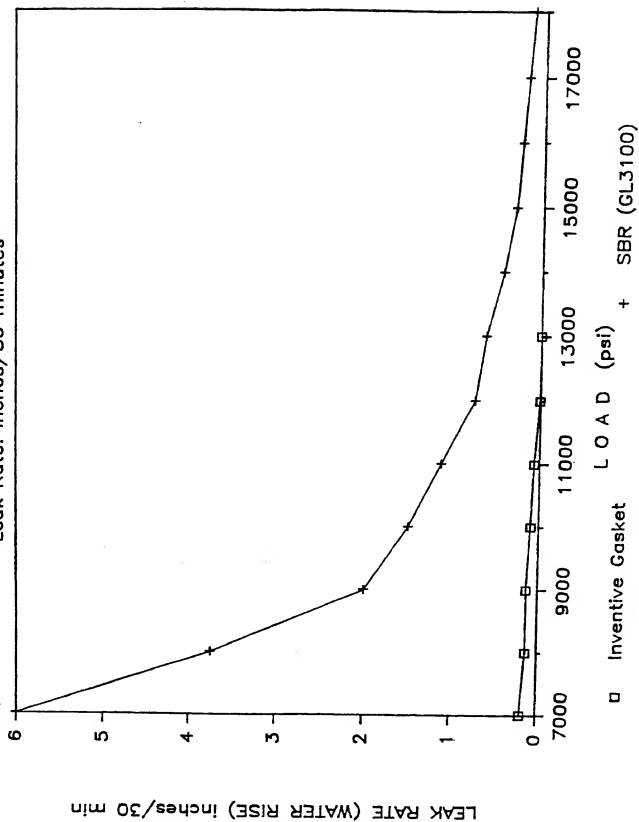
*Fig. 8.* SEALABILITY TEST

Leak Rate: Inches/30 minutes

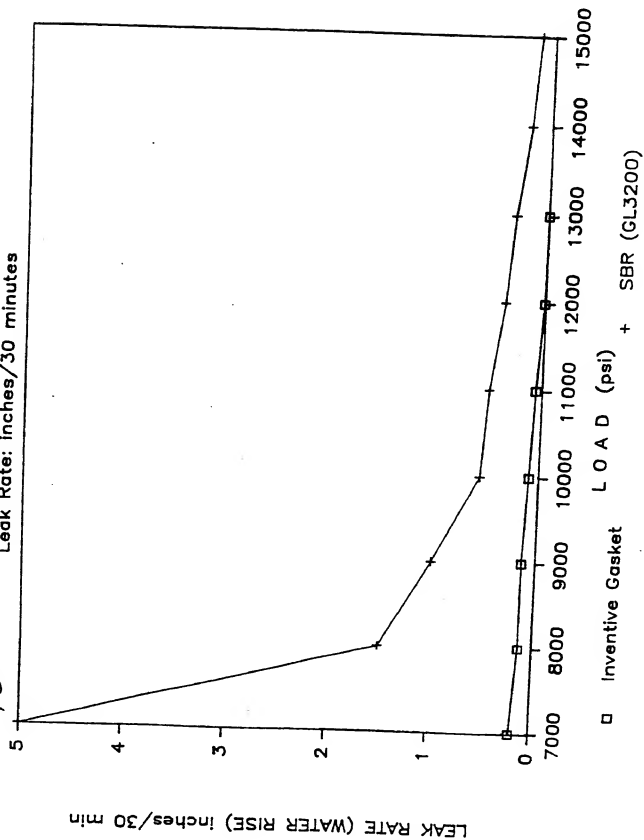


*Fig. 9.* SEALABILITY TEST

Leak Rate: inches/30 minutes

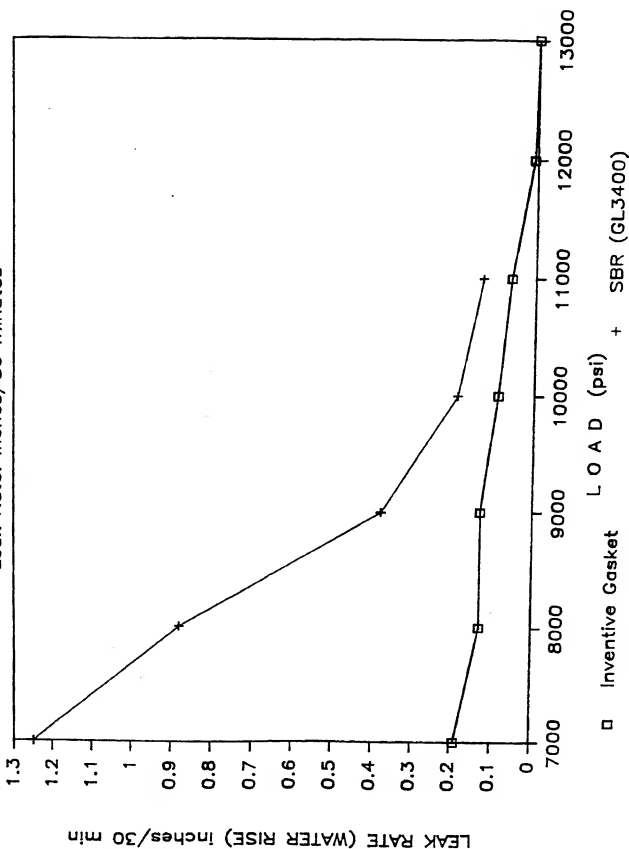


*Fig. 10.* SEALABILITY TEST  
Leak Rate: inches/30 minutes



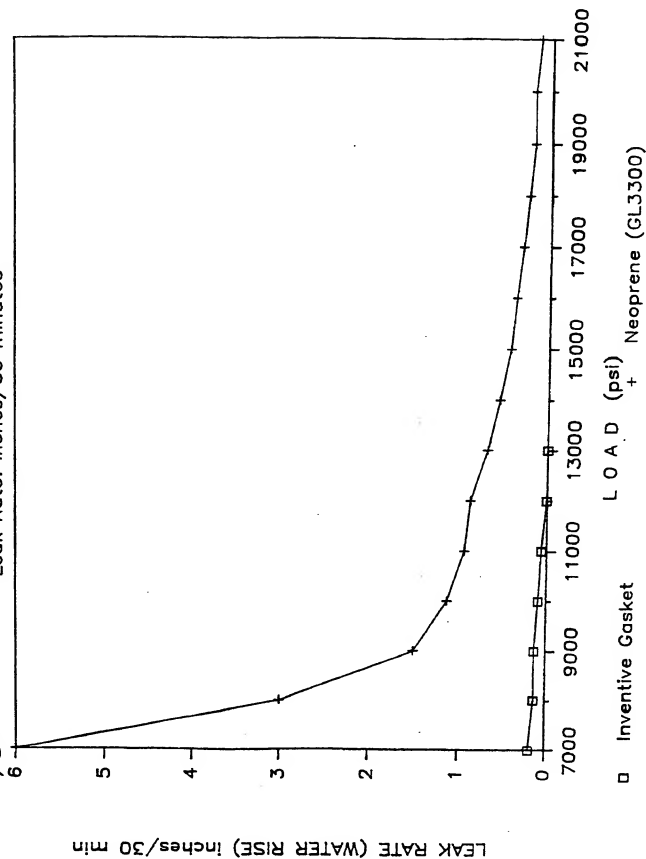
*Fig. 11.* SEALABILITY TEST

Leak Rate: inches/30 minutes



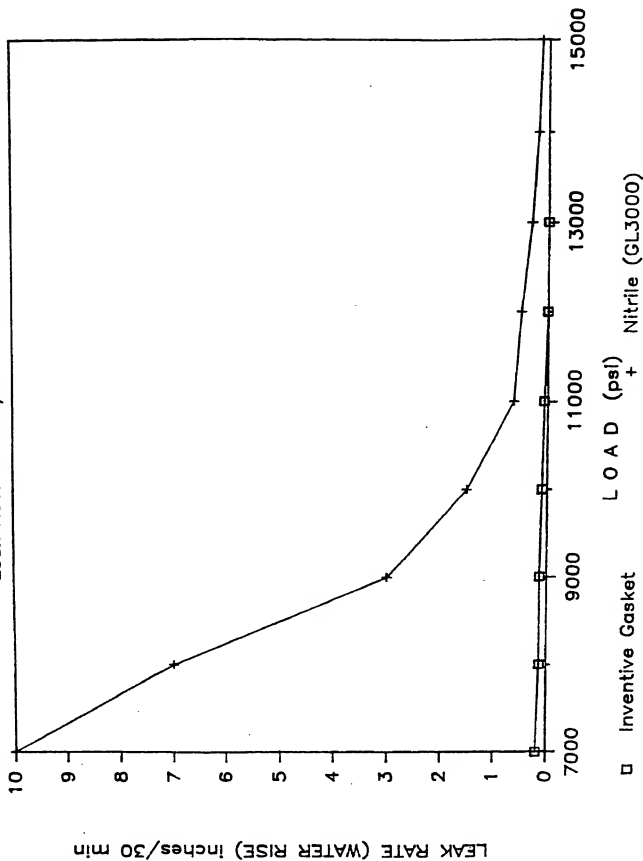
8/11

*Fig. 12.* SEALABILITY TEST  
Leak Rate: inches/30 minutes



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*Fig. 13.* SEALABILITY TEST  
Leak Rate: Inches/30 minutes

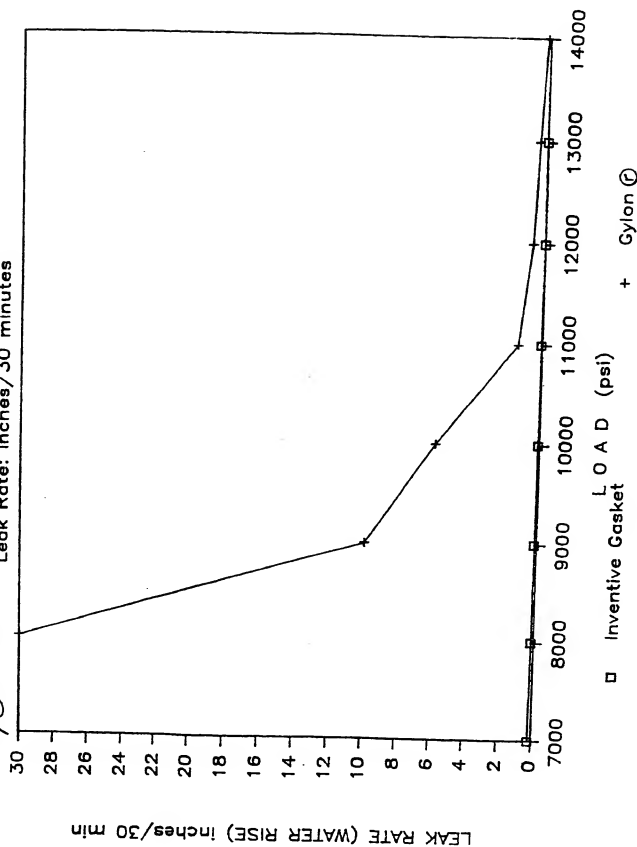




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*Fig. 14.* SEALABILITY TEST

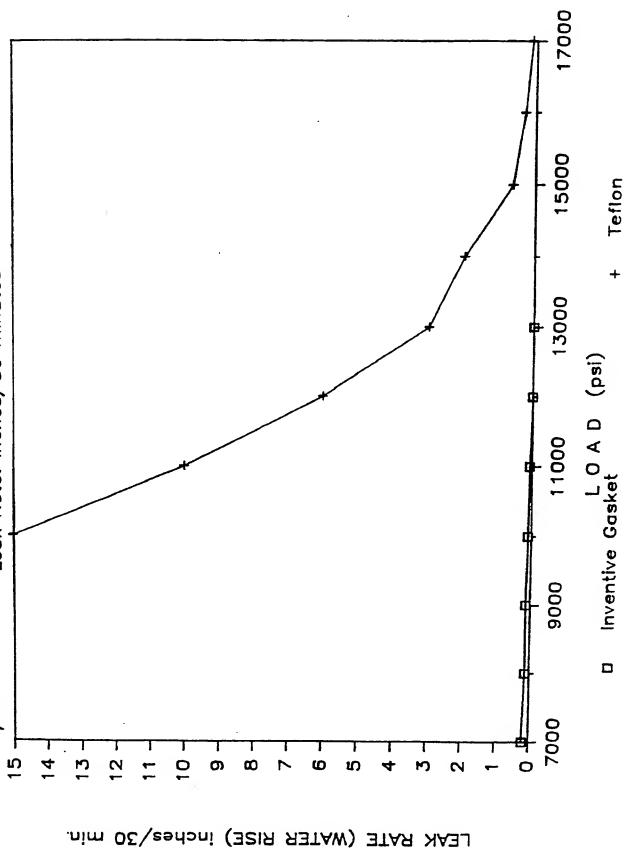
Leak Rate: inches/30 minutes



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*Fig. 15.* SEALABILITY TEST

Leak Rate: inches/30 minutes



# INTERNATIONAL SEARCH REPORT

International Application No PCT/US 89/02302

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (If several classification symbols apply, indicate all) *		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC <sup>4</sup> F 16 J 15/10		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched *		
Classification System	Classification Symbols	
IPC <sup>4</sup>	F 16 J	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched *		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT *</b>		
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	US, A, 4070219 (FARNAM) 24 January 1978, - see figures 4-7; column 4, lines 5-28	1,4,6,23-25, 28,29,31
Y	--	20,21
Y	US, A, 4187390 (GORE) 5 February 1980, see abstract; claims 45-65	20,21
A	cited in the application	2,3,5-18,27, 32
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<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
14th September 1989	21. 10. 89	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	T.K. WILLIS	

# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

US 8902302  
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 02/10/89. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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